OFFICIAL DOCUMENT

Albuquerque Bernalillo County

Water Utility Department

WATER RECLAMATION DIVISION
4201 2ND STREET SW, ALBUQUERQUE, NEW MEXICO 87105

WATER QUALITY LABORATORY
STANDARD OPERATING PROCEDURE APPROVAL FORM

WQL SOP: **227 Total Suspended Solids & Totoal Total Suspended Volatile Solids**

CURRENT REVISION # **04** DATE **May 2006**

ORIGINAL ISSUE DATE **November 1995**

MODIFICATIONS AND REASONS FOR REVISION

*New WQL Management Staff
*New SOP Numbering System
*New QA/QC Filing System
*Revised Quality Control Section

Prepared By: _____ Date: 11 / 1 / 95
Print Name Dave Kersey

Reviewed By: William E. Lindberg Date: 6/5/06
Print Name William E. Lindberg
Laboratory Manager

Reviewed By: Gail Huggins Date: 6/5/06
Print Name Gail Huggins
Quality Assurance Manager

Reviewed By: J. Steven Glass Date: 6/5/06
Print Name J. Steven Glass
Technical Program Manager

Out of Service By: _____ Date: ___/___/___

Reason:

Revision Date: ~~March 2005~~ May 2006

Rev.~~04 3/1/05~~ GAH 5/12/10

STANDARD OPERATING PROCEDURE

SOP 225 ~~225~~ 227

TOTAL SUSPENDED SOLIDS & TOTAL SUSPENDED VOLATILE SOLIDS

SCOPE AND APPLICATION: This method is applicable to drinking, surface and saline water, domestic and industrial wastes. The practical range of determination is 10 mg/L to 20,000 mg/L.

APPLICABLE METHOD REFERENCES:

EPA 160.2 residue non-filterable at 103 to 105°C
18th ed. of Standard Methods, 2540D, E

DISPOSAL OF SAMPLES: Dispose of samples at the sample compositing sink, located in sample preparation area. This sink bypasses the acid trap and goes directly to the sanitary sewer.

Effective Date 6/5/06

TOTAL SUSPENDED SOLIDS

1.0 GENERAL DISCUSSION

1.1 Principle: A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and the total dissolved solids may provide and estimate of the total suspended solids.

1.2 Interference: Exclude large floating particles or submerged agglomerates of non-homogeneous material from the sample if it is determined that their inclusion is not desired in the final result. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to insure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

1.3 Safety Considerations: Samples may be bio-hazardous avoid ingestion, inhalation and contact with the skin. Sample filters are heated to extreme temperatures, avoid contact with the skin or severe burns may result. Use heat resistant gloves, tongs and eye protection when placing or removing filters from ovens or furnaces.

1.4 Sample preservation & Storage: Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. In no case hold sample more than 7 days.

1.5 Sample Preparation: Bring samples to room temperature before analysis.

1.6 Method Performance Criteria: The reference method cites this method as having a standard deviation of 5.2 mg/L (coefficient of variation 33%) at 15 mg/L, 24 mg/L (10%) at 242 mg/L, and 13 mg/L (0.76%) at 1707 mg/L in studies by two analysts of four sets of 10 determinations each. Single-laboratory duplicate analyses of 50 samples of water and wastewater were made with a standard deviation of differences of 2.8 mg/L.

2.0 Apparatus & Equipment: Drying oven, Desiccator (<10 RH), analytical balance (mettler AE-100), wide-mouth pipets, vacuum support manifold, magnetic filtering funnels, magnetic stirrer and magnetic stirring bar, forceps.

2.1 Desiccator Check: The desiccator must be maintained at a Relative Humidity (RH) of <10%. The desiccator RH must be checked once per week via a hygrometer and the check must be documented. Documentation consist of writing the date checked and analyst's initials on a strip of label tape on the side of the desiccator.

2.2 Desiccant renewal: If the RH is >10% then the desiccant must be dehydrated. To dehydrate the desiccant, place the desiccant in an oven at 103 to 105°C for 24 hours.

3.0 Reagents & Supplies: Glass fiber filters (Whatman 934-AH 47mm), disposable aluminum dishes, de-ionized water.

4.0 Quality Control Procedure: Sample replicates are to be conducted on a frequency of one per twenty samples or number five percent of a batch, if the batch comprises more than twenty samples. Replicate samples will have a percent difference calculated and reported at the bottom of the work sheet. Sample batches that have a percent difference greater than 5% must be re-tested. If a second testing of the batch fails to achieve a percent difference less that 5%, that batch of samples will be reported and the data qualified by text on each sample and stating that the percent difference was greater than 5%. A control blank will be conducted on each batch of samples. The control blank must be have a percent difference of less than 5%. If the percent difference is greater than 5%, all samples in the batch are to have the results qualified when reported. Qualify the results by text on the samples and stating that the control blank percent difference was greater than 5%. Results also must be qualified by text on the samples in LIMS, if the drying oven is out of range (103-105°C), or analytical balance won't calibrate. The balance must be checked with S-weights weekly and the results logged in the balance log. Annually the balance must be serviced and calibrated by a certified service person. If the S-weight check is out of the acceptable range (acceptability criteria in balance log) or the balance annual service is not conducted all results subsequent to the last satisfactory S-weight check or one year expiration date of the last annual service of the balance, will be qualified in LIMS by text on the samples.

5.0 Instrument Calibration: Mettler AE-100 Analytical Balance, press and hold the single control bar until **-CAL-** appears in the display, then release control bar. The display changes to **CAL----**, then to **CAL 100** (blinks). Move calibration lever all the way to the rear; the display changes to **CAL----**, followed by 100.0000. Move calibration lever all the way back towards the front of the balance; the display changes to **----**, followed by **0.0000**. If **CAL Err** appears in the display, the weighing pan was not unloaded before calibrating the balance, or the wrong external calibration weight was used (return to the weighing mode by pressing and holding the control bar). **No CAL** appears in the display, a temporary malfunction has occurred (recalibrate balance). Calibration of instrument is to be conducted prior to use.

6.0 Procedure:

6.1 Preparation of glass-fiber filter: Insert filter with wrinkled side up in filtration apparatus. Apply vacuum and wash filter with three successive 20-mL portions of reagent-water. Continue suction to remove filter from filtration apparatus and transfer to an aluminum dish. Take care to prevent the dried filter from adhering to the aluminum dish. Store in desiccator until needed.

6.2 Sample analysis: Using an analytical balance determine the weight of the filter. Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of reagent-grade water to seat it. Stir, sample with a magnetic stirrer, and while stirring, pipet a measured volume onto the seated glass-filter. Wash with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 minutes after

filtration is complete. Samples with high dissolved solids may require additional washings. Carefully remove filter from filtration apparatus and transfer to an aluminum pan as a support. Dry for at least 1 hour at 103-105°C in an oven, cool in a desiccator to balance temperature, and weigh. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Replicate determinations should agree within 5% of their average. If volatile solids are to be determined, treat the residue according to the: **Total Volatile Suspended Solids Procedure.**

7.0 Calculation:

$$\text{mg total suspended solids/L} = \frac{(A - B) \times 1000}{\text{sample volume mL}}$$

where:

A = weight of filter + dried residue, mg.
B = weight of filter, mg.

Conversion of grams to milligrams: grams X 1000 = milligrams

8.0 Reporting: All measurements and results will be recorded in the bound work sheet book for Total Suspended Solids. The determined results for each sample tested will be entered on the electronic data system, SQLLIMS. All samples requiring qualification will be text at the sample level in SQLIMS. All analyses requiring corrective actions will have the documentation of the corrective action in the bound work sheet book, concomitant with the sample results, and QC results.

8.1 Control Charts- All quality control data will be entered in the lab share drive by analysts performing this test. Quality assurance reviews are performed weekly, for complete details of control chart performance evaluations see QA SOP-005.

TOTAL VOLATILE SUSPENDED SOLIDS

1.0 GENERAL DISCUSSION

1.1 Principle: The residue from the Total Suspended Solids procedure is ignited to constant weight at 500 ± 50°C. The remaining solids represent the fixed total, dissolved, or suspended solids while the weight lost on ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater, activated sludge, and industrial wastes.

1.2 Interference: Negative errors in the volatile solids may be produced by loss of volatile matter during drying. Determination of low concentrations of volatile solids in the presence of high fixed solids concentrations may be subject to considerable error.

1.3 Safety Considerations: Samples may be biohazardous avoid ingestion, inhalation and contact with the skin. Sample filters are heated to extreme temperatures, avoid contact with the skin or severe burns may result. Use heat resistant gloves, tongs and eye protection when placing or removing filters from ovens or furnaces.

1.4 Sample preservation & Storage: Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. In no case hold sample more than 7 days.

1.5 Sample Preparation: Bring samples to room temperature before analysis.

1.6 Method Performance Criteria: The reference method cites the standard deviation was 11 mg/L at 170 mg/L volatile total solids in studies by three laboratories on four samples and 10 replicates. Bias data on actual samples cannot be obtained.

2.0 Apparatus & Equipment: Muffle furnace, Desiccator (<10 RH), analytical balance (mettler AE-100), forceps.

3.0 Reagents & Supplies: Glass fiber filters (Whatman 934-AH 47mm), disposable aluminum dishes.

4.0 Quality Control Procedure: Sample replicates are to be conducted on a frequency of one per twenty samples or number five percent of a batch, if the batch comprises more than twenty samples. Replicate samples will have a percent difference calculated and reported at the bottom of the work sheet. Sample batches that have a percent difference greater than 5% must be re-tested. If a second testing of the batch fails to achieve a percent difference less that 5%, that batch of samples will be reported and the data qualified by text on each sample and stating that the percent difference was greater than 5%. A control blank will be conducted on each batch of samples. The control blank must be have a percent difference of less than 5%. If the percent difference is greater than 5%, all samples in the batch are to have the results qualified when reported. Qualify the results by text on the samples and stating that the control blank percent difference was greater than 5%. Results also must be qualified by text on the samples in LIMS, if the drying oven is out of range (103°-105°C), or analytical balance won't calibrate. The balance must be checked with S-weights weekly and the results logged in the balance log. Annually the balance must be serviced and calibrated by a certified service person. If the S-weight check is out of the acceptable range (acceptability criteria in balance log) or the balance annual service is not conducted. All results subsequent to the last satisfactory S-weight check or one year expiration date of the last annual service of the balance, will be qualified in LIMS by placing text on the samples.

5.0 Instrument Calibration: Mettler AE-100 Analytical Balance, press and hold the single control bar until -CAL- appears in the display, then release control bar. The display changes to CAL----, then to CAL 100 (blinks). Move calibration lever all the way to the rear; the display changes to CAL----, followed by 100.0000. Move calibration lever all the way back towards the front of the balance; the display changes to ----, followed by 0.0000. If CAL Err appears in the display, the weighing pan was not unloaded before calibrating the balance, or the wrong external calibration weight was used (return to the weighing mode by pressing and holding the control bar). No CAL appears in the display, a temporary malfunction has occurred (recalibrate balance).

6.0 Procedure:

6.1 Sample analysis: Ignite residue produced by the Total Suspended Solids procedure, to constant weight in a muffle furnace at a temperature of 500 ± 50°C. Have furnace up to temperature before inserting sample. Usually, 15 to 20 minutes ignition are required for 200 mg residue. However more than one sample and/or heavier residues may overtax the furnace and necessitate longer ignition times. Let dish and filter cool partially in air until most of the heat has been dissipated. Transfer to a desiccator for final cooling in a dry atmosphere. Do not overload desiccator. Weigh filter as soon as it has cooled to balance temperature. Repeat cycle of igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% or 0.5 mg, whichever is less.

7.0 Calculation:

$$\text{mg volatile solids/L} = \frac{(A - B) \times 1000}{\text{sample volume mL}}$$

mg fixed solids/L = $\frac{(B - C) \times 1000}{\text{sample volume mL}}$

where:

A = weight of residue + filter before ignition, mg.
B = weight of residue + filter after ignition, mg, and
C = weight of filter, mg.

8.0 Reporting: All measurements and results will be recorded in the bound work sheet book for Total Suspended Solids. The determined results for each sample tested will be entered on the electronic data system, SQLLIMS. Replicate determinations should agree within 5% of their average.

8.1 Control Charts- All quality control data will be entered in the lab share drive by analysts performing this test. Quality assurance reviews are performed weekly, for complete details of control chart performance evaluations see QA SOP-005.